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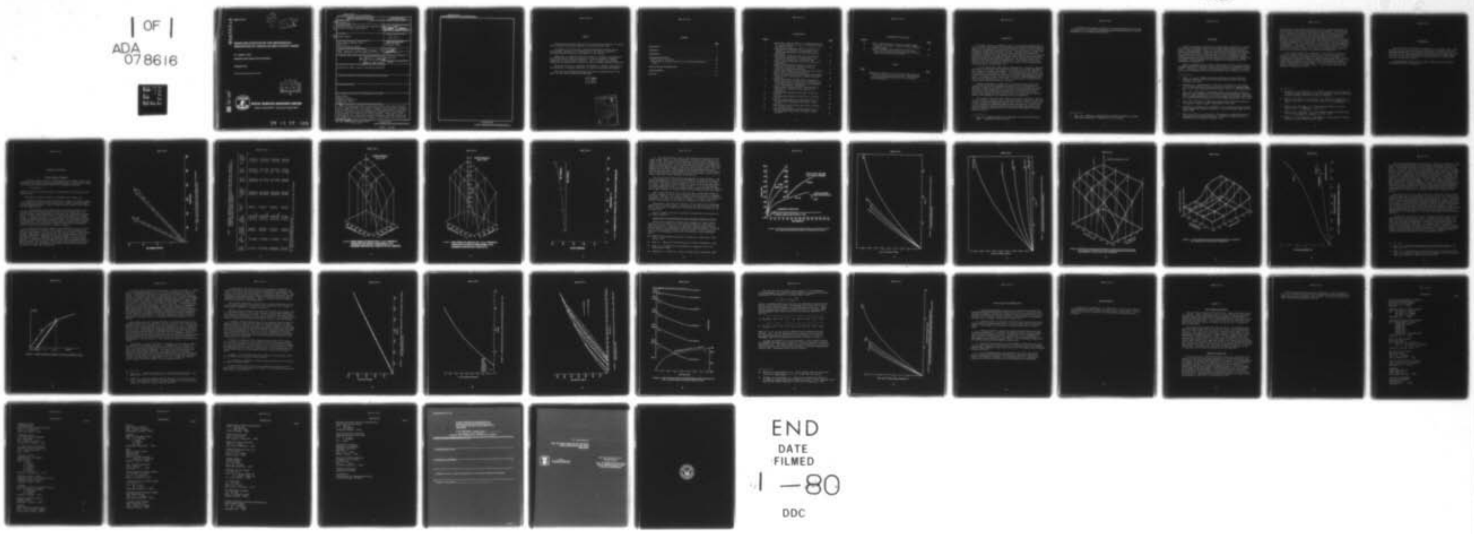

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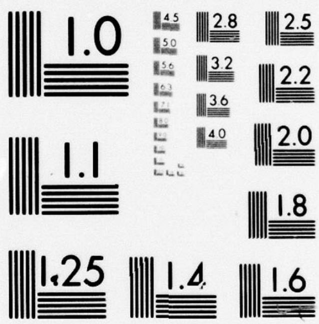

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NSWC TR 79-41

MOISTURE EFFECTS ON THE MECHANICAL PROPERTIES OF HERCULES 3501-6 EPOXY RESIN

BY JOSEPH M. AUGL

RESEARCH AND TECHNOLOGY DEPARTMENT

30 MARCH 1979

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REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER
14 NSWC/TR-79-41

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)
6 Moisture Effects on the Mechanical Properties of Hercules 3501-6 Epoxy Resin.

5. TYPE OF REPORT & PERIOD COVERED
9 Progress Report.
Oct[redacted]77-Oct[redacted]78,

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)
10 Joseph M./Augl

8. CONTRACT OR GRANT NUMBER(s)
12 45

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Naval Surface Weapons Center
White Oak
Silver Spring, MD 20910

391 596

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
61153N; WR 02204;
WR 0220401; R31AA

11. CONTROLLING OFFICE NAME AND ADDRESS
16 WR02204, WR22000

12. REPORT DATE
11 30 Mar[redacted]79

13. NUMBER OF PAGES
42

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)
17 WR0220401
WR2200000

15. SECURITY CLASS. (of this report)
UNCLASSIFIED

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release, distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Epoxy Resin
Moisture Effect
Mechanical Properties
Temperature Effect

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The following properties of the Hercules 3501-6 epoxy resin were determined: Young's modulus, shear modulus, Poisson's ratio, ultimate strength, and ultimate strain, all as a function of temperature (between 21° and 150°C) and as a function of moisture concentration corresponding to an equilibrium concentration from 0-100 percent relative humidity. The purpose of this investigation was to obtain the necessary constituent material property data for predicting the environmental response of 3501-6 carbon fiber reinforced composites.

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SUMMARY

The epoxy resin Hercules 3501-6 will be used as matrix material for carbon fiber reinforced structural components in advanced Navy aircraft.

The purpose of this work was to determine mechanical baseline data of this material, necessary for the prediction of the composite response to projected material and service environments of the aircraft.

These data, in conjunction with the concepts of diffusion, micromechanics and laminate theory, allow the prediction of certain changes in composite properties such as the elastic constants. Also, it appears that strength changes may be predicted as a function of time for various climactic conditions.

This work is part of a continuing investigation to predict quantitatively the degradation of composite materials in a service and storage environment.

This work was sponsored by the Naval Air Systems Command during FY 1978 under the Task number A3200000/004A/9R2200000.

J. R. Dixon

J. R. Dixon
By direction

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INTRODUCTION

This effort should be considered as part of a continuing investigation to obtain a better understanding of the property deterioration of fiber reinforced organic matrix composites in quantitative terms as a function of their service environment during their projected life time. The high strength to weight and stiffness to weight ratios of fiber reinforced composites have long been recognized as benefits for light weight structural materials and it is therefore natural that they are, although not without caution and certain reservations, being introduced as major structural materials into military aircraft such as the F-16, the future F-18 and the AV8B Harrier. The primary driving factor though for the use of composite materials in aircraft is the projected potential saving due to reduced labor cost.

It is well known that organic matrix composites are, in general, affected by moisture in that they lose strength and stiffness at elevated temperatures after exposure to ambient environments. For safety reasons, this requires a certain degree of over design. Thus it is not unusual that materials are used for practical applications long before they are fully characterized and understood. As a matter of fact, a great deal of material understanding has come from practical applications, or at least, the practical use of materials has lead to further fruitful scientific scrutiny.

Various micromechanical schemes such as finite difference and finite element methods have been recognized as powerful tools in describing the mechanical properties of composites from their constituent properties (resin and fiber). These methods allow to calculate not only macroscopic stress-strain fields over several ply thicknesses but also microscopic stress-strain fields between and inside single fibers. All these theories require, however, the knowledge of the constituent material properties.

The purpose of this work was to obtain such material properties, specifically, the elastic constants (Young's and shear modulus, Poisson's ratio), the ultimate strength and elongation together with the total stress strain curves, all as a function of temperatures and moisture concentration. The moisture sorption equilibria and the temperature and concentration dependance of the moisture diffusion coefficient of the resin will be discussed in a separate Technical Report.[1]

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1. Augl, J.M., "Moisture Sorption and Diffusion in Hercules 3501-6 Epoxy Resin," NSWC/WOL TR 79-39 (in print).

The prediction of composite properties from the experimental data of this investigation will likewise be compared with experimentally measured composite properties in a following Technical Report.[2]

2. Augl, J.M., "Prediction and Verification of Moisture Effects on Carbon Fiber Epoxy Composites," NSWC/WOL TR 79-43 (in print).

BACKGROUND

Aside from excessive, mechanical or thermal loading conditions, it appears that moisture will perhaps lead to the most significant property changes in organic matrix composites at least for the matrix dominated lamina properties such as intralamina shear, transverse tensile and both transverse and longitudinal compressive properties. Since moisture will have to penetrate into the composite to affect its properties and since the rate of penetration depends on the temperature and on the moisture concentration these property changes will be gradual, strongly dependent on the thickness of the composite and on the environment it is exposed to.

Several investigators have presented experimental evidence that the matrix dominated properties of advanced organic matrix composites are degraded by moisture[3-13]. This effect increases as the temperature of the composite is raised[6]. We have also demonstrated that the property deterioration levels out after the

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3. Hertz, J., et al., "Advanced Composite Application for Spacecraft and Missiles," AFML-TR-71-186, Vol II, General Dynamics-Convair, San Diego, California, Mar 1972.

4. Browning, C.E., and Whitney,J.E., "The Effect of Moisture on the Properties of High Performance Structural Resins and Composites," American Chem. Soc., Div. of Org. Coatings and Plastic Chem., 33, No. 2, 1973, pp. 137-148.

5. Augl, J.M., "Environmental Degradation Studies on Carbon Fiber Reinforced Epoxies," Paper presented at the TTCP-Panel P3 Meeting Melbourne, Australia, 1975, and paper presented at the Air Force Workshop on Durability Characteristics of Resin Matrix Composites, Battelle-Columbus Laboratories, Ohio, 1975.

6. Augl, J.M., "The Effect of Moisture on Carbon Fiber Reinforced Composites. II Mechanical Property Changes," NSWC/WOL/TR 76-149, 1977.

7. Browning, C.E., Husman, G.E., and Whitney, J.M., "Moisture Effects in Epoxy Matrix Composites," Composite Materials: Testing and Design, ASTM STP 617, 1976, p. 481.

8. Hofer, K.E., Rao, N., and Larson, D., "Development of Engineering Data on the Mechanical and Physical Properties of Advanced Composite Materials," AFML-TR-72-205, IIT Research Institute, 1974.

absorbed moisture has reached equilibrium with the surrounding humidity and is, therefore, a function of the average relative humidity. This deterioration is reversible for matrixes that are not chemically changed by moisture (as is the case for most amine cured epoxy composites where moisture is thought only to plasticize the resin and thus make it more flexible[5,6]). Note that it is not only the glass transition temperature that is lowered but the entire modulus and the stress-strain curves are affected far below glass transition temperature and increasingly so as the glass transition temperature is approached. This is important for the understanding of the deterioration of properties in composites. Under reversible property changes it is meant that if the moisture is removed (in vacuum or by heating) the original properties will be regained within the experimental uncertainties.

It may require decades for a composite to reach moisture equilibrium with the environment during which time there is a continuous change in properties over time and throughout the thickness of the composite. Thus a quantitative prediction of composite property changes must include at least a simultaneous treatment of moisture diffusion, micromechanics, laminate theory, and modeling of the environment, to be able to deal with daily and seasonal temperature and humidity changes. A more detailed discussion has been given in References 1 and 2.

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9. Browning, C.E., and Hartness, J.T., "Effect of Moisture on the Properties of High Performance Structural Resins and Composites," Composite Materials: Testing and Design, ASTM STP 546, 1974, pp. 284-302.

10. Kaelble, D.H., Dynes, P.J., and Crane, L.W., "Interfacial Mechanisms of Moisture Degradation in Graphite-Epoxy Composites," J. of Adhesion, 7, 25 1977.

11. Kaelble, D.H., and Dynes, P.J., "Surface Energy Analysis of Treated Graphite Fibers," J. Adhesion, 6, 239, 1974.

12. Ashbee, K.H.G., and Wyatt, R.C., "Water Damage in Glass Fiber/Resin Composites," Proc. Royal Soc., Series A, 312, 533, 1969.

13. Halpin, J.C., and Pagano, N.J., "Consequences of Environmentally Induced Dilation in Solids," AFML-TR-68-395, 1969.

EXPERIMENTAL

The resin 3501-6 was obtained from Hercules Inc. (Bacchus, Utah). After degasing the resin was cast into sheet form and cured. Dogbone specimens were machined and fitted with biaxial strain gages. After moisture equilibration at 0, 30, 55, 80, and 100 percent relative humidity the samples were tested at 21°, 60°, 100°, and 150°C on an Instron Universal Tensile Tester for their stress-strain behavior.

The experimental details for the sample preparation and the property measurements are given in Appendix A.

DISCUSSION AND RESULTS

Initial Elastic Constants

The initial elastic constants (Young's modulus and Poisson's ratio) were determined with biaxial strain gage measurements (Fig. 1) from which the shear modulus was calculated from the relation for homogeneous materials:

$$G = E/[2(1+\nu)]$$

(where G, E and ν are the shear modulus, Young's modulus, and Poisson's ratio respectively).

The results are given in Table 1 and graphically in Figures 2-4.

It should be noted here that the use of strain gages in measuring organic resins properties requires special consideration. Great care must be taken to avoid measurement errors. Special attention shoud be given to the following experimental details:

1. Since plastics are bad heat conductors the heat generated in the resistance wire of the gage during the initial current surge leads to an initial change in local temperature (at the gage site) indicating an apparent strain. It takes about 30-40 seconds to reach equilibrium after the input voltage is applied. Therefore, it is not advisable to switch back and forth between the longitudinal and the transverse gage. The measurements should be run subsequently. The strains should not exceed the elastic limit in order to prevent errors in the subsequent transverse stress-strain measurements. A typical initial stress-strain curve is shown in Figure 1.

2. While the strain gages adhered to the specimen well over the whole temperature range up to fracture on dry samples or at low temperatures on moisture loaded samples, this was not the case for the moisture loaded samples at higher temperature. The bond between resin and gage broke well before fracture, which made it impossible to use strain gages to obtain the total elastoplastic range of temperature and humidity effects for this material. Therefore a combination of strain gage and strain gage extensometer measurements were used. The low strain, initial moduli were obtained with the more accurate strain gage readings while the complete stress-strain curves were obtained with strain gage extensometers.

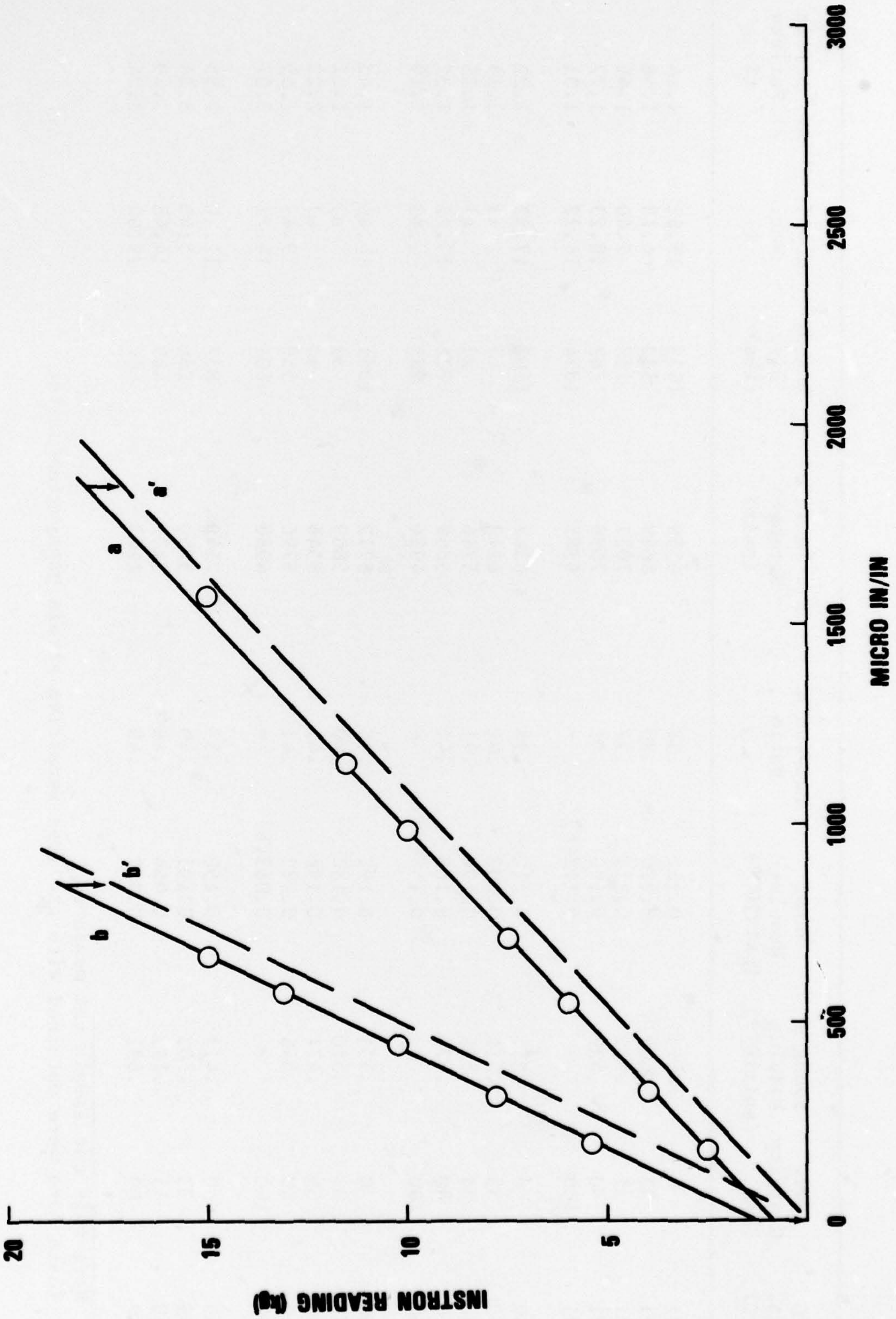

FIGURE 1 TYPICAL RUN : HERCULES 3501-6, a) LONGITUDINAL STRAIN b) TRANSVERSE STRAIN : a' AND b' ARE CORRECTED TO GO THROUGH ZERO

TABLE 1 - MECHANICAL PROPERTIES OF HERCULES 3501-6 EPOXY RESIN AS A FUNCTION OF TEMPERATURE AND MOISTURE (CORRESPONDING TO THE EQUILIBRIUM CONCENTRATION OF THE INDICATED RELATIVE HUMIDITY)

Test Temp. (° C)	Rel. Humidity (%)	Young's Modulus ($psix10^{-6}$)	Shear Modulus ($psix10^{-6}$)	Poisson's Ratio	Av. Tensile Strength (psix)	Stand. Div. (lbs)	Coeff. Var. (%)	Av. Strain At Failure (%)
21	0	.615	0.222	.38	6559	1053	15.82	1.14
21	33	.609	0.220	.38	8649	530	6.13	1.56
21	55	.582	0.211	.37	7027	168	2.40	1.46
21	80	.529	0.190	.41	7698	787	10.23	1.77
21	100	-	0.173(b)	-	6285	1054	16.77	1.31
60	0	.585	0.212	.38	6367	1103	17.33	1.22
60	33	.516	0.203	.41	6243	a)	a)	1.13
60	55	.536	0.190	.41	5746	a)	a)	1.35
60	80	.478	0.168	.41	5869	776	13.22	1.32
60	100	-	0.150(b)	-	4984	281	5.63	1.19
100	0	.531	0.192	.38	6712	1301	18.40	1.43
100	33	.510	0.182	.40	5809	a)	a)	1.22
100	55	.471	0.168	.45	8546	a)	a)	2.22
100	80	.346	0.122	.41	5796	550	9.49	1.59
100	100	-	0.083(b)	-	4088	601	14.71	3.07
150	0	.417	0.150	.39	7649	857	11.20	2.55
150	33	.302	0.101	.49	5250	286	5.45	3.34
150	55	.197	0.066	.49	3239	469	14.48	3.49
150	80	.081	0.027	.49	2283	662	29.02	3.24

a) Here only one sample was measured
b) These data were obtained with the less sensitive strain gage extensometer

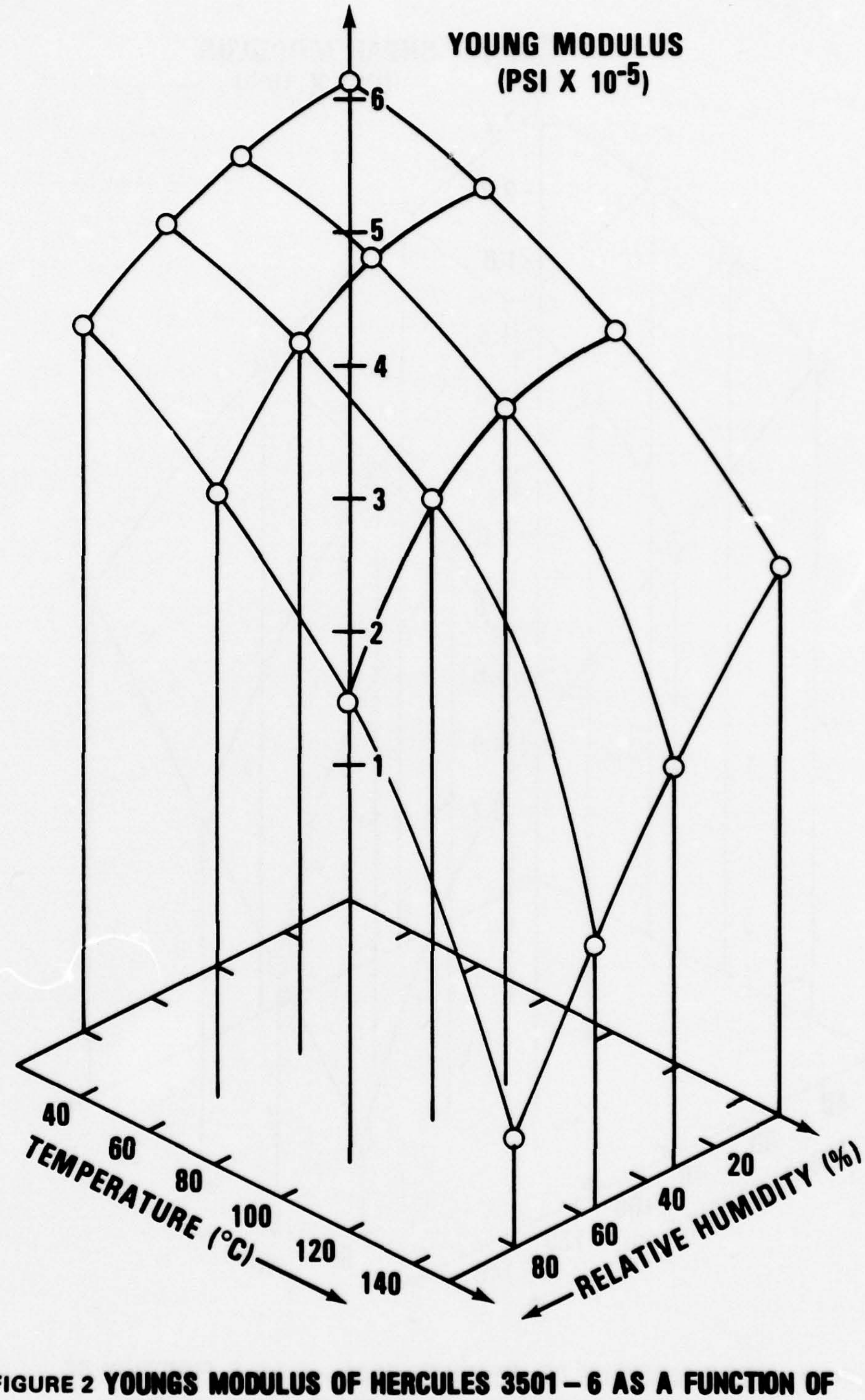

FIGURE 2 **YOUNGS MODULUS OF HERCULES 3501 – 6 AS A FUNCTION OF TEMPERATURE AND MOISTURE (CORRESPONDING TO THE EQUILIBRIUM CONCENTRATION OF INDICATED RELATIVE HUMIDITIES)**

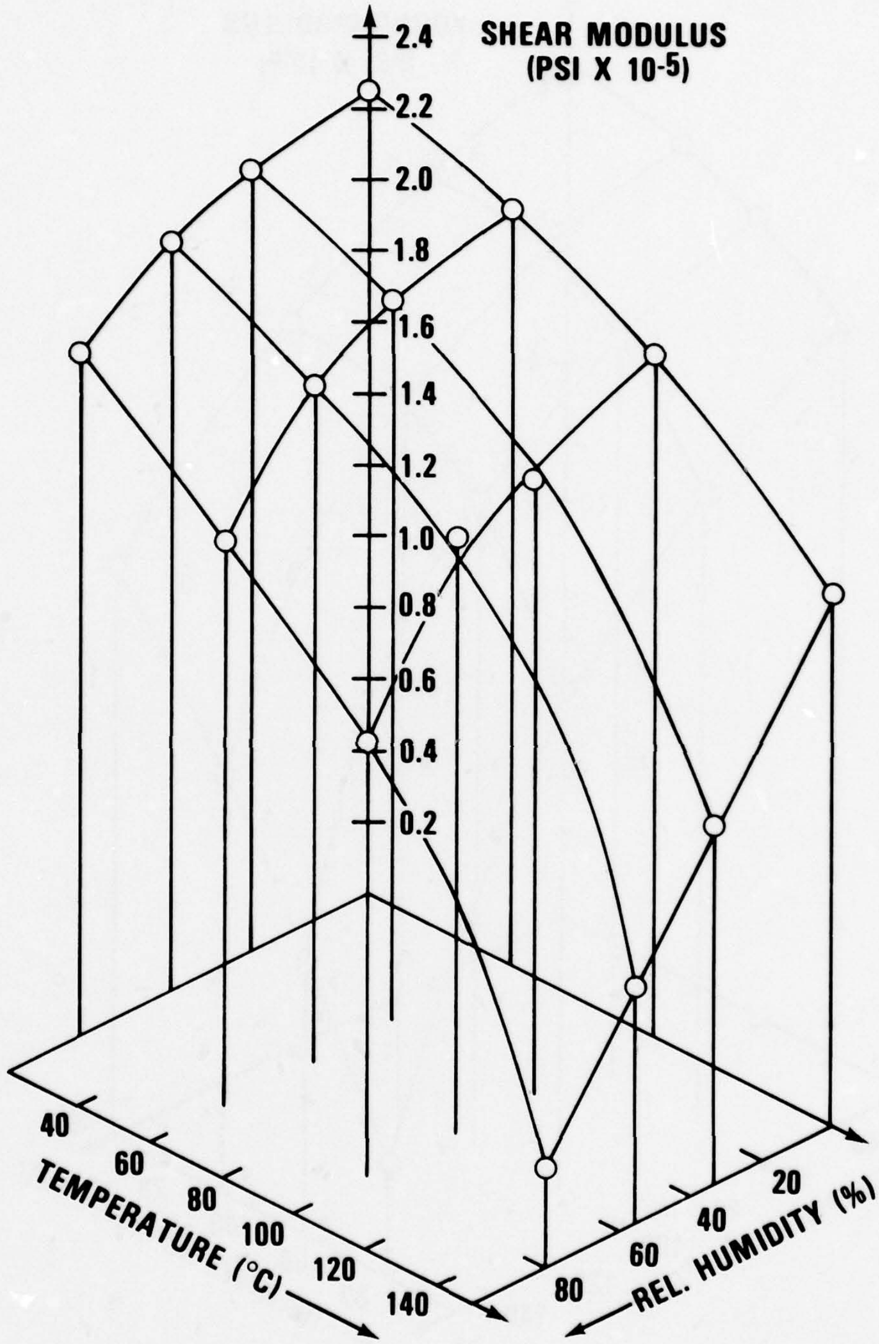

FIGURE 3 **SHEAR MODULUS OF HERCULES 3501– 6 AS A FUNCTION OF TEMPERATURE AND MOISTURE (CORRESPONDING TO THE EQUILIBRIUM CONCENTRATION OF INDICATED RH)**

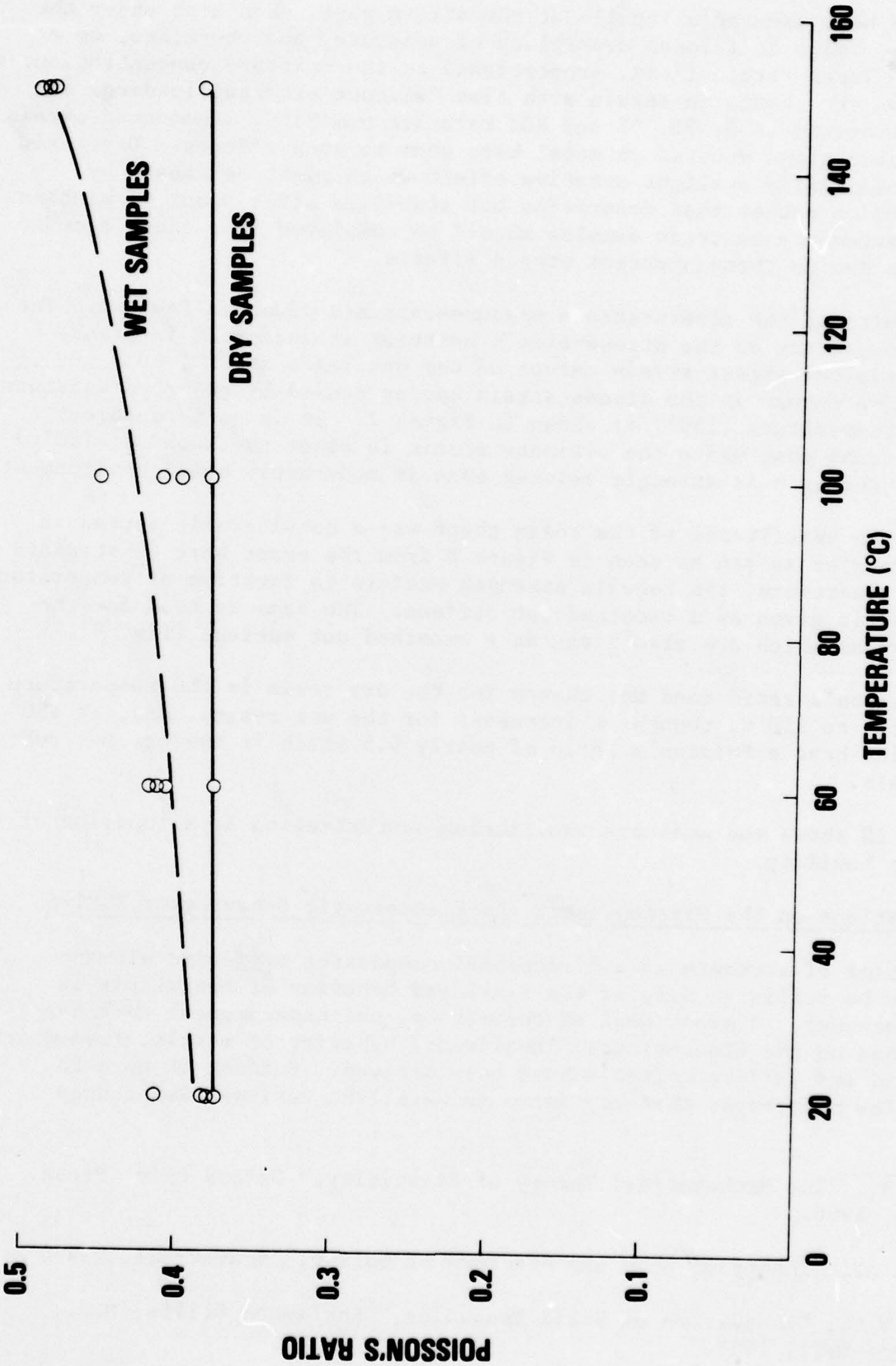

FIGURE 4 POISSON'S RATIO OF HERCULES 3501-6 AS A FUNCTION OF TEMPERATURE

3. The heat generated locally at the strain gage, when kept under the input voltage, leads to a local desorption of moisture, and therefore, to a change in the local strain field, proportional to the moisture concentration. Figure 5 shows the change in strain with time (without external loading) for samples equilibrated at 0, 33, 55 and 80% relative humidity. Unmounted strain gages and strain gages mounted on metal bars show no such effects. Dry resin samples show initially a slight negative effect which might be caused by moisture sorption rather than desorption but stabilize after about 40 minutes. Thus the measurements on resin samples should be completed in a short time to reduce errors due to these apparent strain effects.

The results of the stress-strain measurements are given in Table 1. The effect of temperature on the stress-strain behavior is indicated in Figure 6 which shows typical stress-strain curves of the dry resin at 21°, 60°, 100°, and 150°C. The change in the stress-strain curves caused by absorbed moisture at constant temperature (150°) is shown in Figure 7. It is quite apparent from these graphs that while the ultimate strain is about the same (at 150°C) the ultimate strength is strongly reduced even in moderately humid environments.

Due to the brittleness of the resin there was a considerable spread in ultimate strengths as can be seen in Figure 8 from the error bars ($\pm$ standard deviation). Therefore, the tensile strength profile (a function of temperature and humidity) is given as a smoothed out surface. The same is true for the ultimate strains which are also given as a smoothed out surface (Fig. 9).

The Poisson's ratio does not change for the dry resin in the temperature range from 21° to 150°C, though it increases for the wet resins, and, at 150° the wet resins have a Poisson's ratio of nearly 0.5 which is typical for rubbery materials.

Figure 10 shows the moisture equilibrium concentration as a function of the relative humidity.

Observations on the Elastoplastic and Viscoelastic Behavior of 3501-6

Prediction of strength in undirectional composites by finite element analysis can be realistic only if the nonlinear behavior of the matrix is taken into account. A great deal of theoretical and experimental work has been published on the elastoplastic (nonlinear) behavior of metals, from which certain yield and failure criteria have been derived. Suffice it here to refer to a few monographs that may serve as excellent reviews (References 14-17).

14. Hill, R., "The Mathematical Theory of Plasticity," Oxford Univ. Press. London, 1950.

15. Nadai, A., "Theory of Flow and Fracture of Solids," McGraw-Hill, 1950.

16. Fung, Y.C., "Foundation of Solid Mechanics," Englewood Cliffs, N.J., Prentice-Hall, 1965.

17. Mendelson, A., "Plasticity: Theory and Application," Macmillan, 1968.

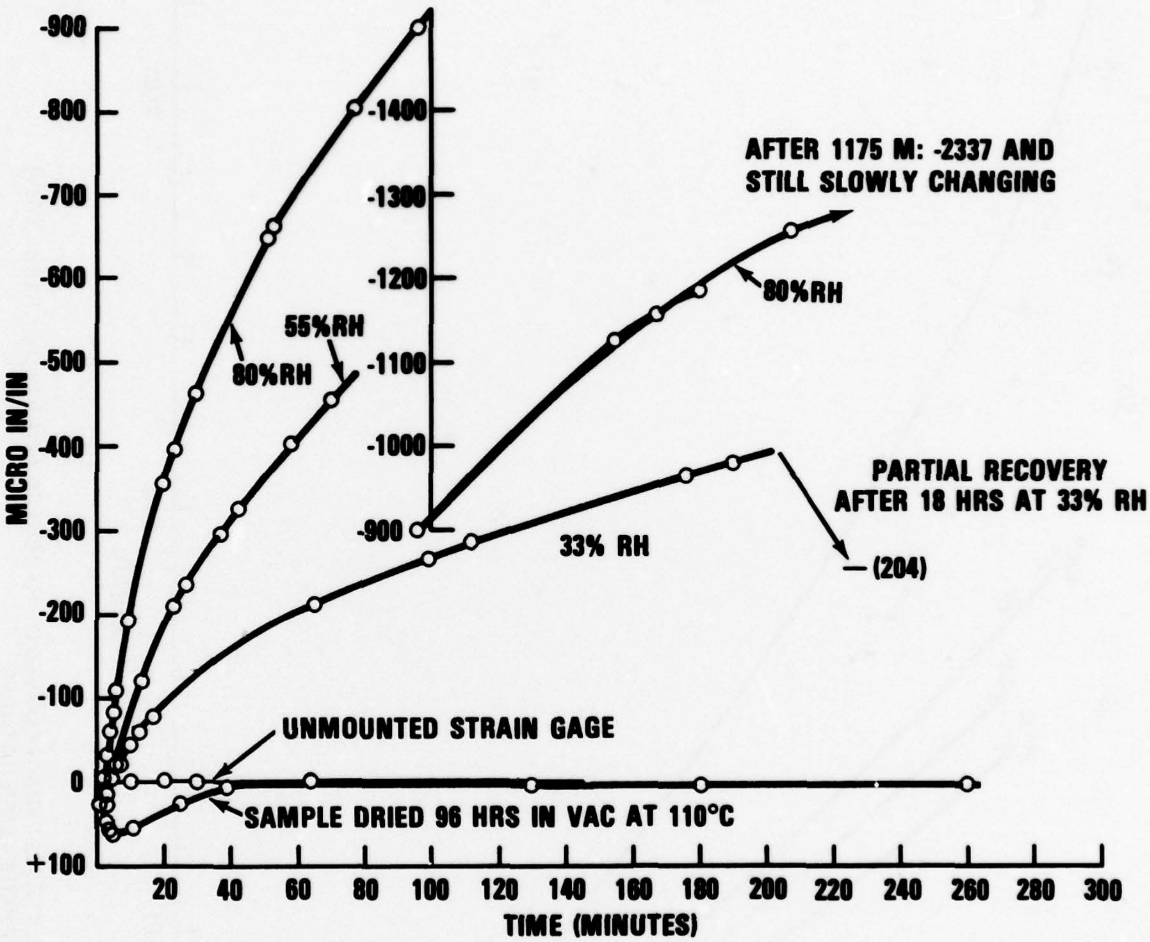

FIGURE 5 APPARENT STRAIN MEASURED IN HERCULES 3501–6 AS A FUNCTION OF TIME DUE TO MOISTURE DESORPTION (CAUSED BY THERMOCOUPLE HEATING)

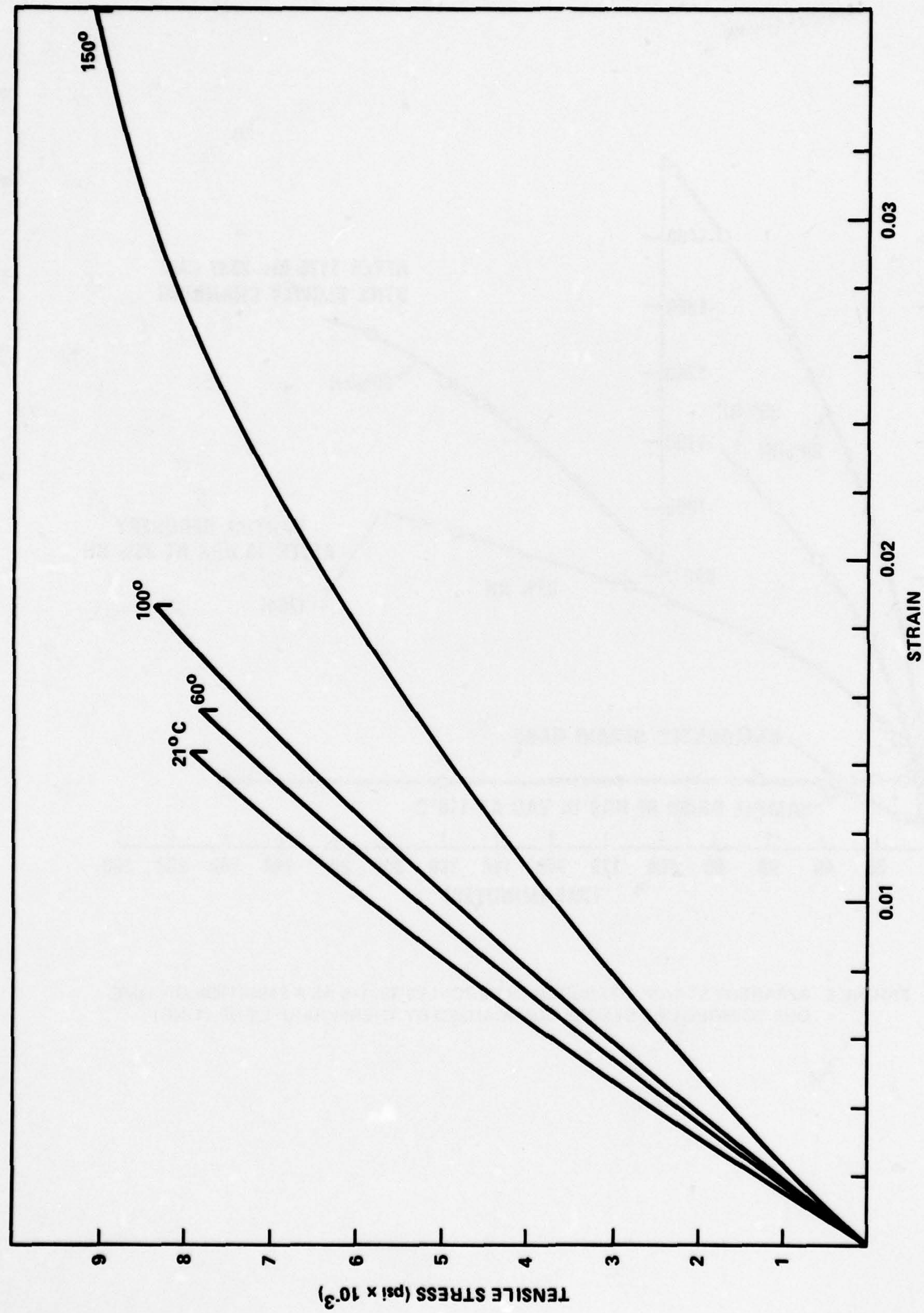

FIGURE 6 TYPICAL STRESS STRAIN CURVES OF HERCULES 3501-6 EPOXY RESIN (DRY) AT DIFFERENT TEMPERATURES

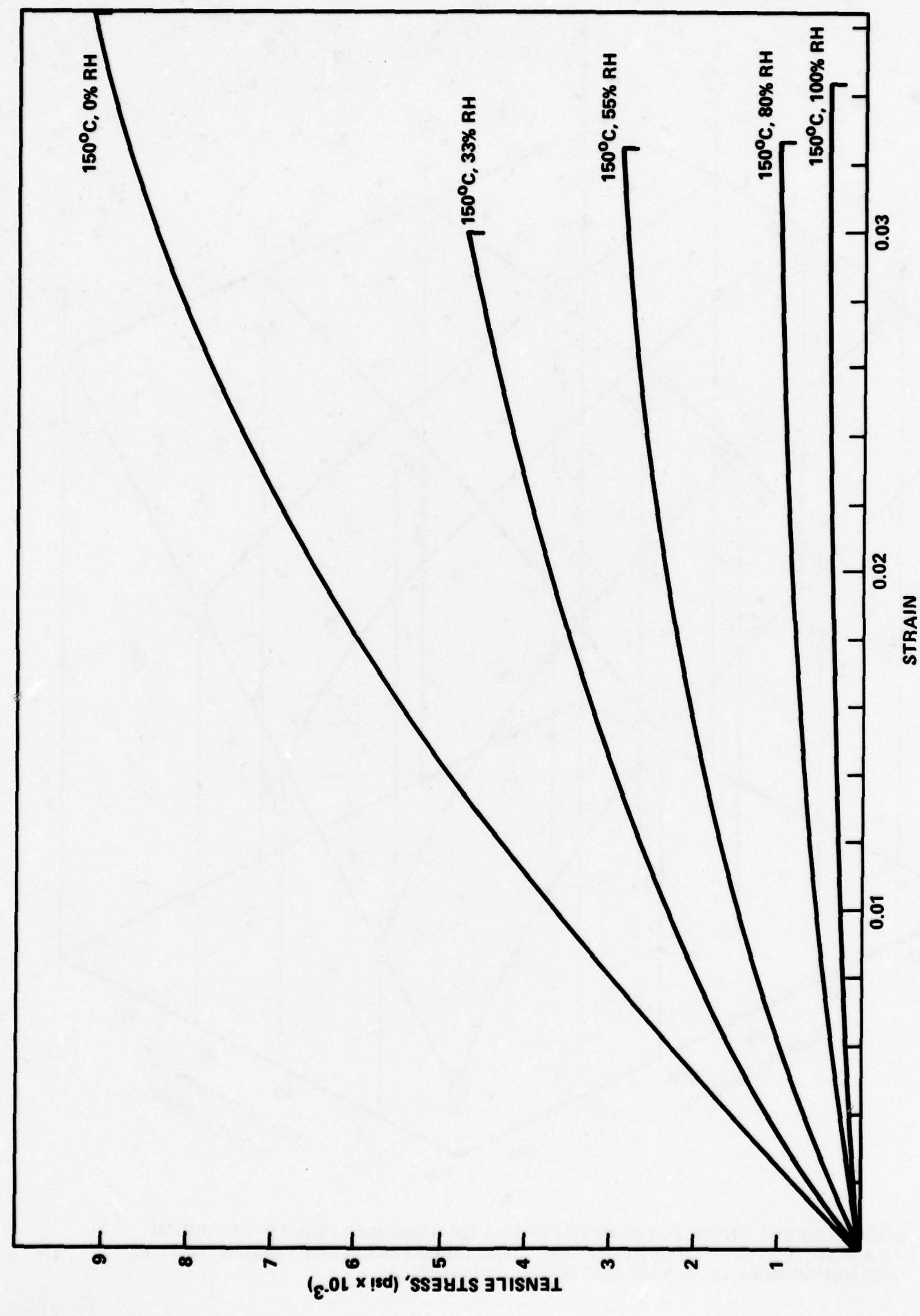

FIGURE 7 TYPICAL STRESS STRAIN CURVES OF HERCULES 3501-6 EPOXY RESIN (AT 150°C) EQUILIBRATED AT DIFFERENT RELATIVE HUMIDITIES

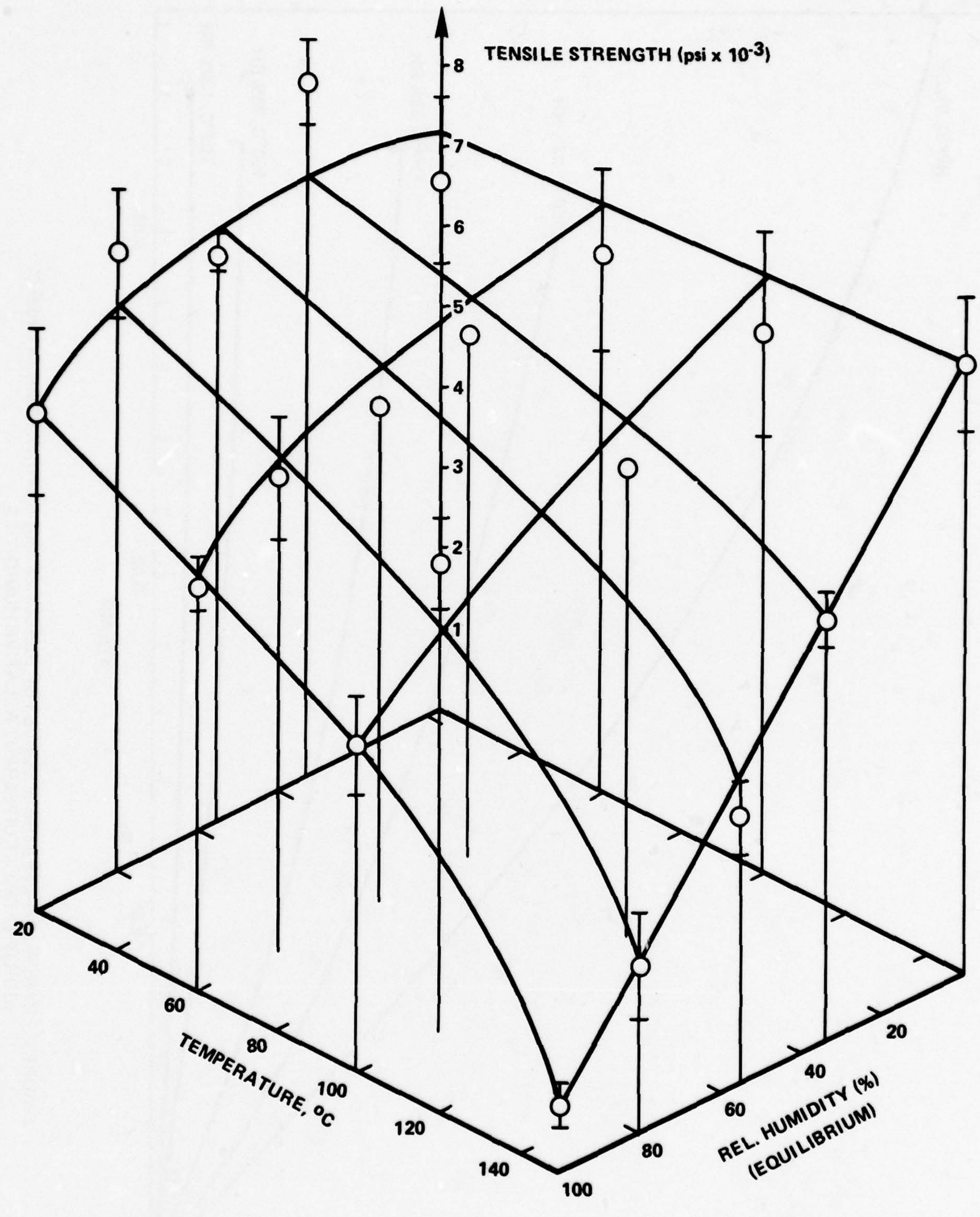

FIGURE 8 SMOOTHED OUT TENSILE STRENGTH PROFILE OF HERCULES 3501-6 EPOXY RESIN AS A FUNCTION OF TEMPERATURE AND EQUILIBRIUM MOISTURE CONCENTRATION (CORRESPONDING TO THE INDICATED REL. HUMIDITIES

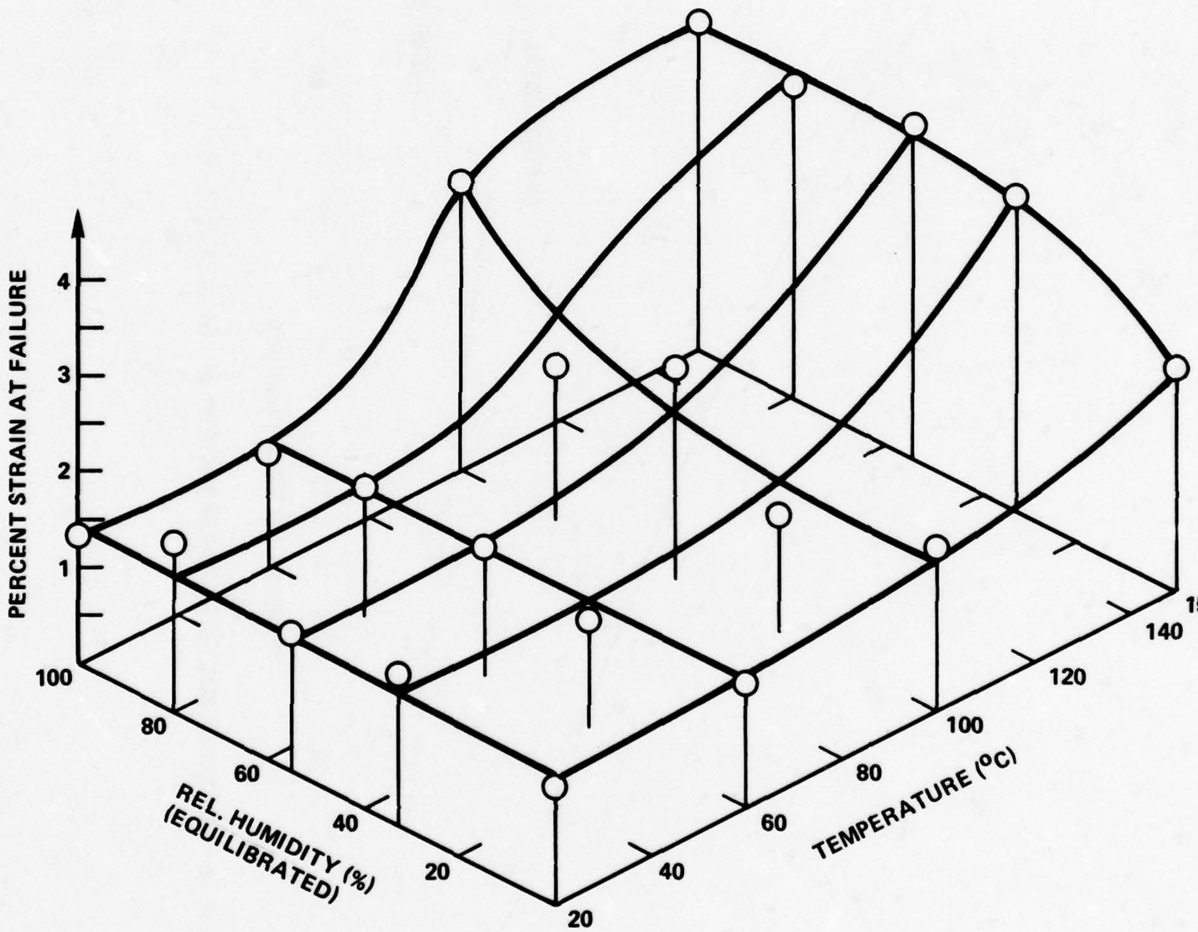

FIGURE 9 FAILURE STRAIN OF HERCULES 3501-6 (EQUILIBRATED AT DIFFERENT REL. HUMIDITIES) AS A FUNCTION OF TEMPERATURE

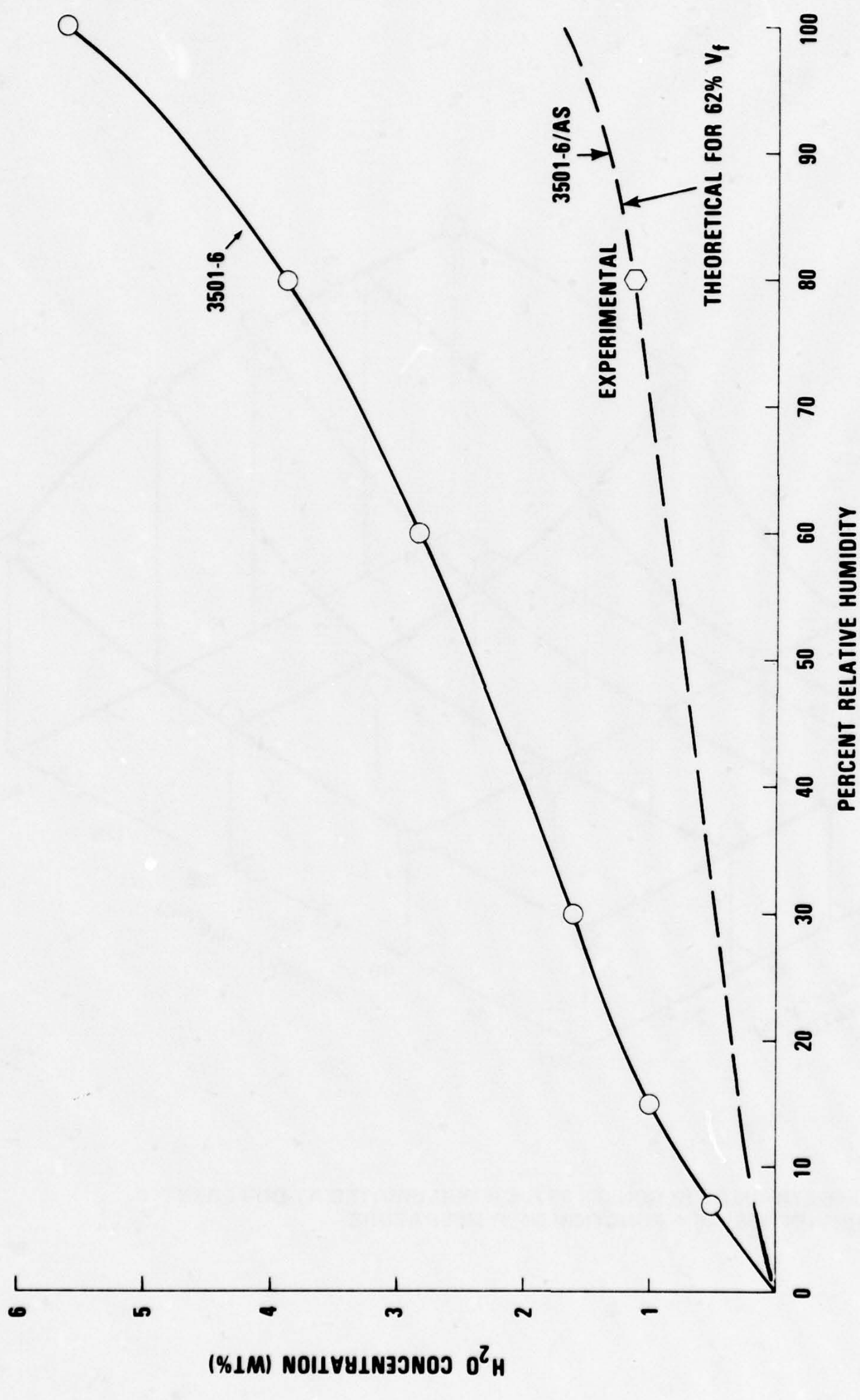

FIGURE 10 EQUILIBRIUM CONCENTRATION OF MOISTURE IN HERCULES 3501-6

The stress-strain behavior of most metals is indicated in Figure 11. They show a linear stress-strain curve till the stress reaches point 1 (the yield point). An additional increase in stress leads to a nonlinear increase in strain. If at point 2 the stress is released there is now a linear unloading, the slope of which is equal to the initial modulus, thus, leading to a residual strain at zero stress which is a permanent plastic deformation. Renewed loading will show a linear stress strain behavior (following the unloading curve) till point 2 is reached which is now the new yield point. In metallurgy this behavior is called strain hardening. For most metals there is no deformation which is both load and time dependent. Thus a finite element treatment of metal matrix fiber reinforced composites can describe the nonlinear behavior of these materials by a stepwise incremental loading procedure were experimental stress-strain data are used for the current constituent stiffness parameters of the respective elements[18, 19]. The three dimensional state of stress was described by using the concept of octahedral shear stress-octahedral shear strain.

Some authors have used the same formalism in dealing with organic matrix composites which may not be justified, at least not in all cases, because many polymers are not only elastoplastic but also viscoelastic, i.e., a viscous flow may occur under an applied load, which upon unloading, may, wholly, partially, or not recover. This difference in mechanical behavior of polymers and metals can be easily understood from the different molecular structure. While metals are essentially lattices made of atoms which are held together by electric forces, ploymers are molecular chains which atoms are covalently bonded together. These polymer chains may interact with others by weak Van der Waals forces (thermoplastic, linear polymers) or with crosslinks (rubbers and thermosetting resins).

A deformation in metals may lead to a slippage of crystal lattice planes and therefore to a permanent deformation. A deformation in a polymers can lead to a stretching of polymer chains, which can be restored if there is a sufficient inter chain entanglement. In rubbers the crosslinks prevent the chains from sliding apart, therefore, the original shape is essentially recovered if none of the chains have been broken during the straining process. However, even in rubbers the restoring process may be slowed down considerably or may be prevented when the temperature is too low to supply the required thermal energy (activation energy, for chain segment mobility).

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18. Cook, R.D., Concepts and Applications of Finite Element Analysis, John Wiley, 1974.

19. Adams, D.F., "High-Performance Composite Materials for Vehicle Construction: An Elastoplastic Analysis of Crack Propagation in a Unidirectional Composite," Rand Corp., Report R1070-PR, Mar 1973.

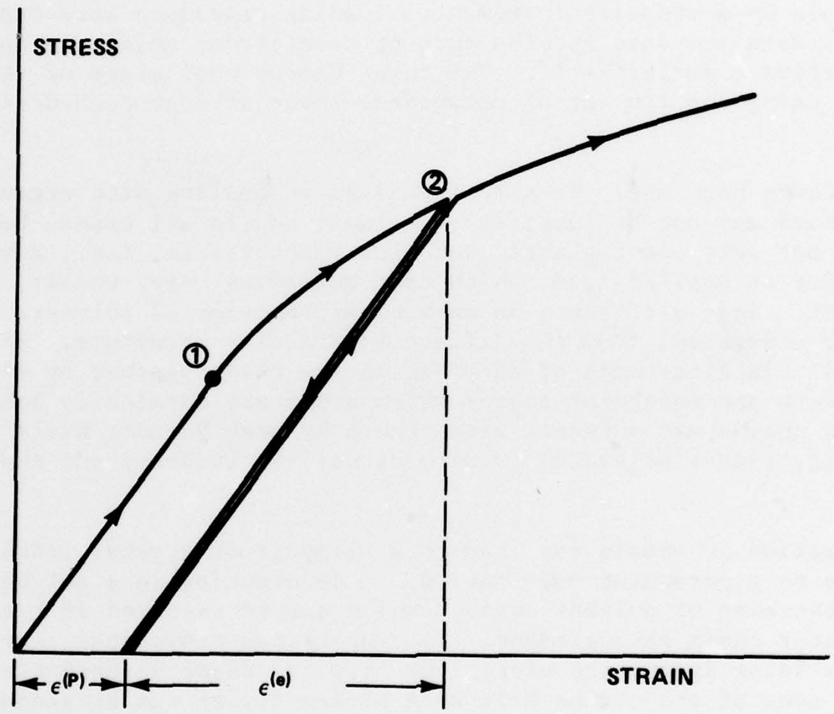

FIGURE 11 STRAIN-HARDENING (CHANGE IN YIELD POINT IN UNIAXIAL TESTS).

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19. Adams, D.F., "High-Performance Composite Materials for Vehicle Construction: An Elastoplastic Analysis of Crack Propagation in a Unidirectional Composite," Rand Corp., Report R1070-PR, Mar 1973.

In other words, the viscous flow is quite temperature dependent. It increases strongly as the glass transition temperature is approached from below. At the same time the modulus of the polymer decreases rapidly. Thus, prediction of the stress strain behavior in composites, using only the time independent displacement fields is valid only at temperatures sufficiently below glass transition temperature. What is sufficient should be experimentally determined by creep measurements.

The following experiments were not made to obtain quantitative data for the viscoelastic behavior of the 3501-6 resin but rather to see whether it would follow a behavior as indicated in Figure 11.

Figure 12 shows a loading (solid line) and unloading curve (dashed line) of a dry 3501-6 specimen. Only a small plastic or viscoelastic deformation was observed over the range of strain. At 125°C however, the plastic deformation was quite substantial as can be seen in Figure 13. The reloading curve however, showed still the same initial modulus it had at the first loading.

A repeated loading-unloading curve with increasingly higher stresses is shown in Figure 14. Here only the lowest stress application (up to about 3000 psi) showed the behavior of Figure 11; at higher stresses the unloading and reloading curves did not follow the same paths, in addition, at zero load there was a slow partial recovery of the plastic deformation which increased with increasing deformation. The fact, that the reloading and unloading curves do not follow the same path can be easily explained by a (time dependent) viscoelastic deformation since the stress-strain measurements are not instantaneous but rather slow.

To test for viscoelastic flow a sample was maintained at 125° while a static load was increased in steps from 20 to 95 kg as indicated in Figure 15. Each load increment was held constant for five minutes while the change in strain was measured. As can be seen from the set of curves (solid lines) the deformation rate increases with applied load. After unloading (at 1 kg residual load) there was a slow partial recovery (indicated by the broken line).

It appears, that at temperatures above 100°C the time dependent properties of 3501-6 can no longer be safely neglected.

It is therefore, advisable to include creep measurements for the characterization of Hercules 3501-6.

As was mentioned above, for the three dimensional description of the elastroplastic stress-strain field the concept of the octahedral shear stress-shear strain is very useful.[19]

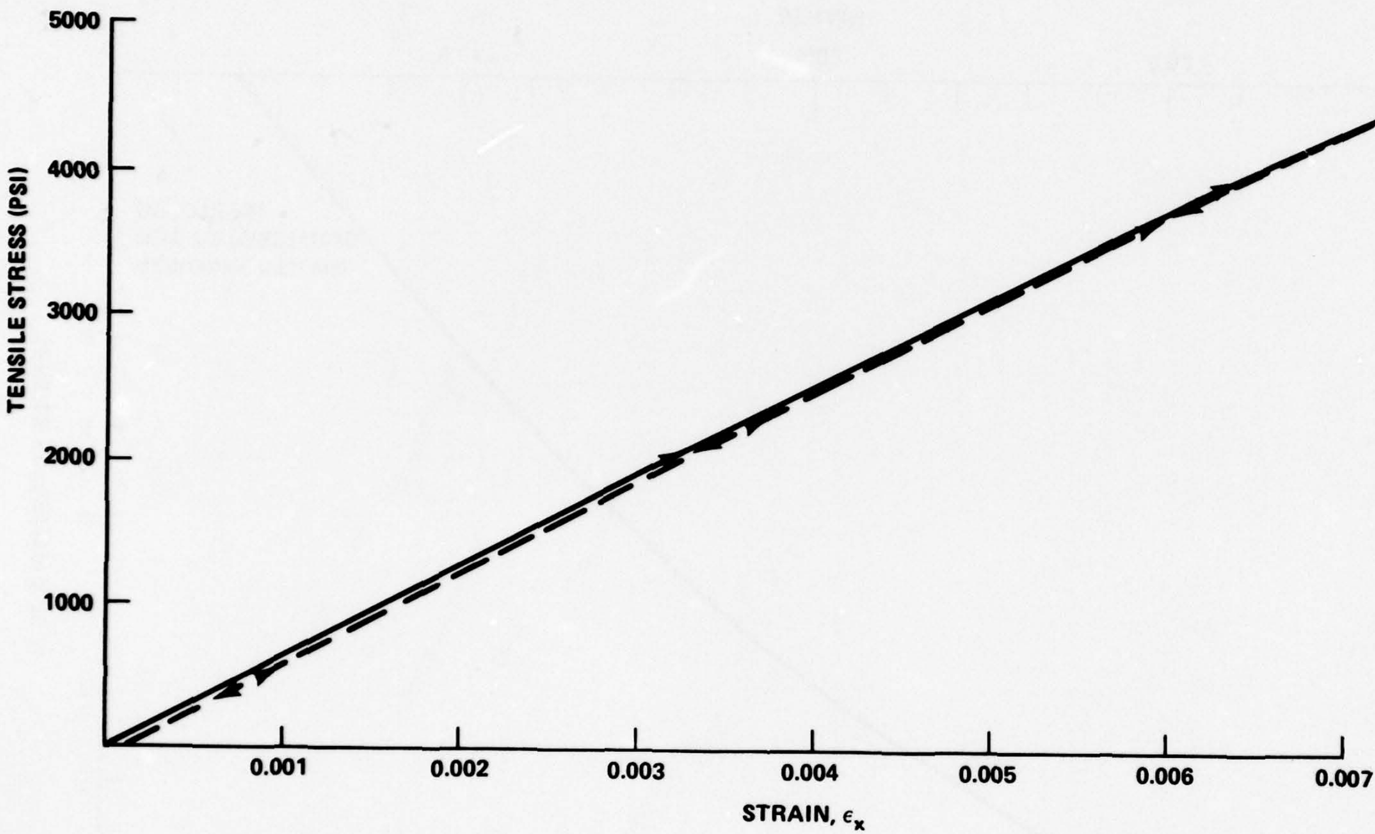

FIGURE 12 TEST FOR ELASTO-PLASTICITY AND VISCOELASTICITY IN HERCULES 3501–6 EPOXY RESIN (DRY RESIN, TESTED AT 21°C).

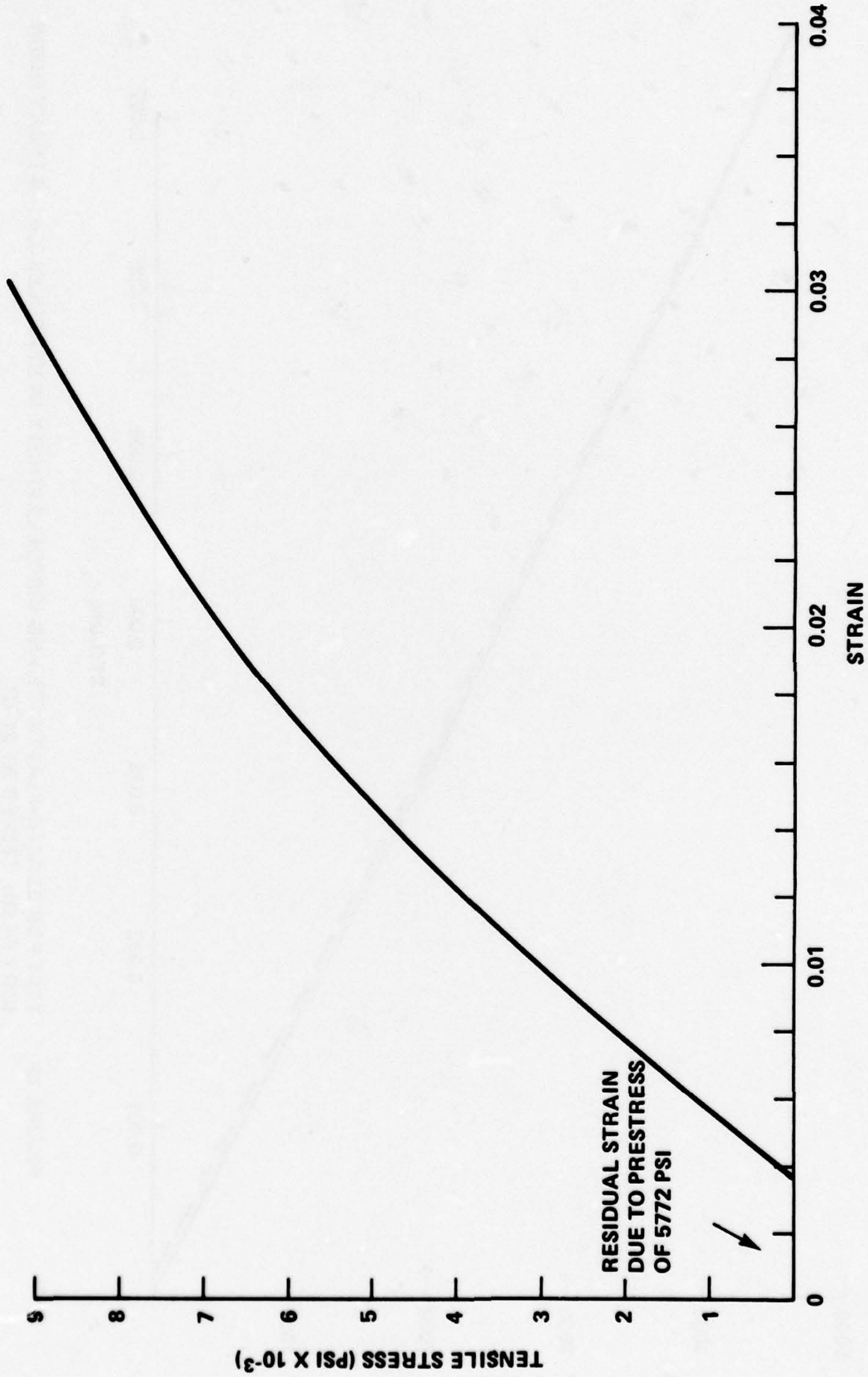

FIGURE 13 STRESS STRAIN CURVE OF 3501–6 RESIN AFTER PRESTRESS TO 5772 PSI AT 125°C.

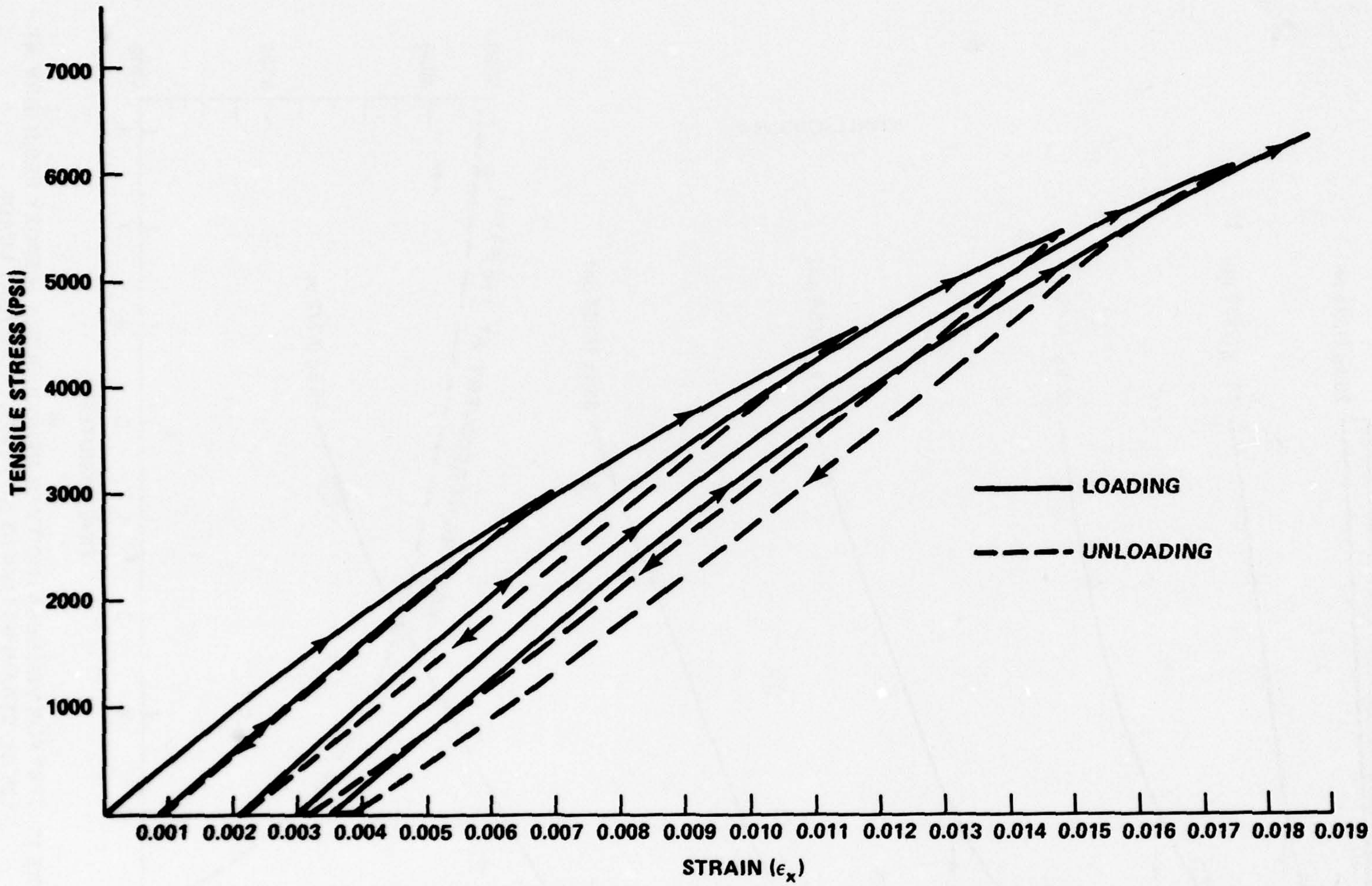

FIGURE 14 TEST FOR ELASTO-PLASTICITY AND VISCOELASTICITY IN HERCULES 3501–6 EPOXY RESIN (DRY RESIN, TESTED AT 125°C).

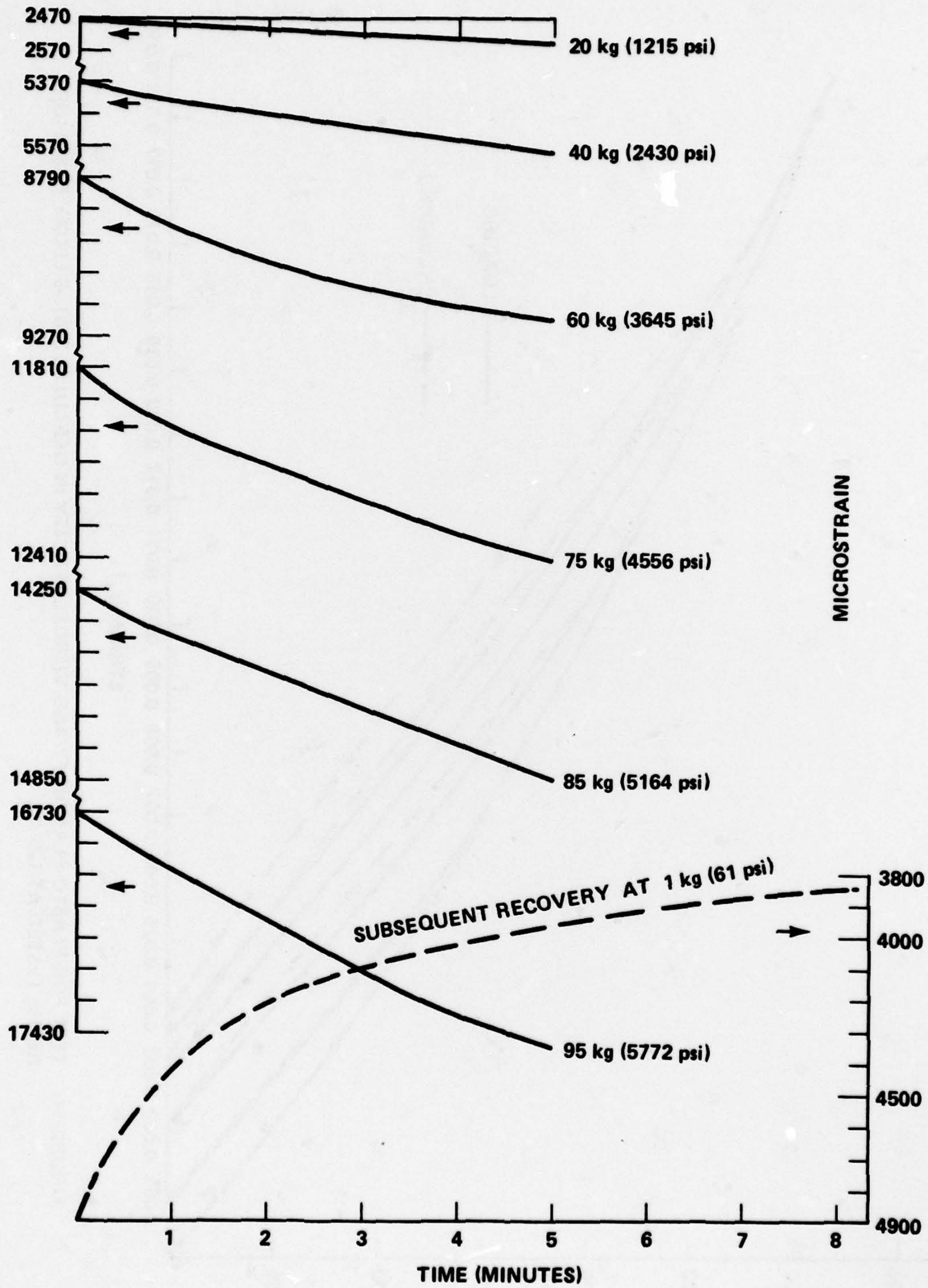

FIGURE 15 TEST FOR VISCOELASTICITY OF HERCULES 3501–6 EPOXY RESIN (DRY, AT 125°C). STRAIN VS TIME AT DIFFERENT CONSTANT LOADS.

The input data for the finite element analysis of the experimental stress-strain data can be given by a two parameter curve proposed by Richard and Blacklock[20] and modified for octahedral shear stress[19]:

$$\tau_o = \bar{E}\bar{\varepsilon}/[1 + |\bar{E}\bar{\varepsilon}/\sigma_o|^n]^{\frac{1}{n}}$$

where τ_o = octahedral shear stress $\bar{\sigma}_o$ and n are independent parameters which fit the curve to empirical data, $\bar{\varepsilon}$= octahedral shear strain, $\bar{E}$= slope of initial portion of the octahedral shear stress-shear strain curve. Simple tensile stress-strain curves can be readily converted to octahedral shear stress-octahedral shear strain curves from the relations (Reference 15, p. 103, 115):

$$\tau_o = \frac{1}{3}\sqrt{(\sigma_X - \sigma_Y)^2 + (\sigma_Y - \sigma_Z)^2 + (\sigma_Z - \sigma_X)^2 + 6 \quad \tau_{XY}{}^2 + \tau_{YZ}{}^2 + \tau_{ZX}{}^2)}$$

$$\bar{\varepsilon} = \frac{2}{3}\sqrt{(\varepsilon_X - \varepsilon_Y)^2 + (\varepsilon_Y - \varepsilon_Z)^2 + (\varepsilon_Z - \varepsilon_X)^2 + \frac{3}{2} \quad \gamma_{XY}{}^2 + \gamma_{YZ}{}^2 + \gamma_{ZX}{}^2}$$

where σ_X, σ_Y, σ_Z, ε_Y, ε_Z, are the respective axial stresses and strains and γ_{XY}... γ_{XY}... are the respective shear stresses and shear strains. As a typical example the stress-strain curves of Figure 6 in terms of octahedral shear stress-octahedral shear strain are shown in Figure 16.

Crossman and Flaggs[21] have recently reported a finite element analysis for composite laminates which includes the viscoelastic behavior of the unidirectional plies. This method appears to be more promising for the description of composites (with high moisture loadings or at elevated temperatures) than the time - independent elastic-plastic analysis.

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20. Richard, R.M. and Blacklock, J.R., "Finite Element Analysis of Inelastic Structures," AIAA Journal, Vol. 7, No. 3, Mar 1969, pp. 432-438.

21. Crossman, F.W. and Flaggs, D.L., "Prediction of Dimensional Stability of Composite Laminates during Environmental Exposure," Paper presented at the 24th National SAMPLE Symposium, 8-10 May 1979, Proceedings, p. 998.

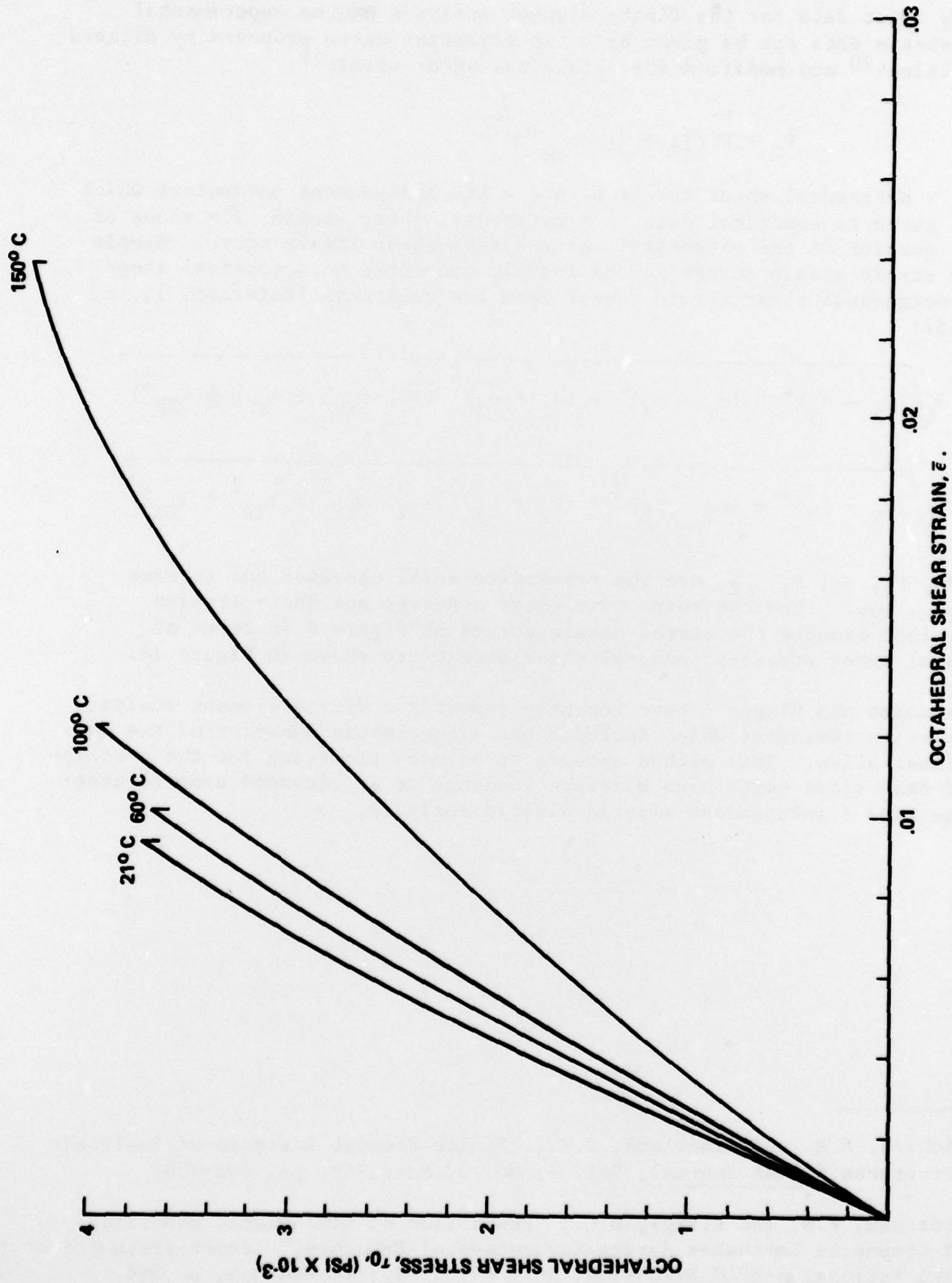

FIGURE 16 TYPICAL OCTAHEDRAL SHEAR STRESS - OCTAHEDRAL SHEAR STRAIN CURVES OF DRY HERCULES 3501-6 EPOXY RESIN (DERIVED FROM TENSILE STRESS - STRAIN CURVES).

CONCLUSIONS AND RECOMMENDATIONS

1. The following mechanical properties of Hercules 3501-6 epoxy resin have been determined from tensile stress-strain curves as a function of temperature and moisture equilibrium concentration in the range from 21°C to 150° and from 0 to 100 percent relative humidity: Young's modulus, shear modulus, Poisson's ratio, ultimate strength, and ultimate strain.

2. Herclues 3501-6 is a brittle resin which has a fairly linear stress-strain behavior at ambient temperatures, with an ultimate strain of about 1.5 percent. The ultimate strain increases to slightly more than 3 percent at 150°C.

3. Neither strength nor modulus is significantly affected by moisture at ambient temperatures. At elevated temperatures, however, moisture affects both, modulus and strength, severely. For example, the dry strength of the resin at 150°C is 7650 psi while the strength of the resin equilibrated at 100 percent relative humidity is only 790 psi. The corresponding Youngs moduli are 417000 psi and less than 20000 psi respectively.

4. Above 100° the viscoelastic (time dependent) deformation under load can no longer be neglected and will have to be considered for a full composite analysis where temperatures above 100°C play a role (for example under supersonic heating conditions).

5. It is recommended that creep experiments be carried out on both resin and composites at different temperatures to establish a "WLF Master Curve". For composite specimens it is recommended that ±45° laminates be used which will allow to determine the time dependent shear deformation.

ACKNOWLEDGEMENTS

I would like to thank Messrs. M. Stander and C. Bersch of the Naval Air Systems Command for their support and their interest in this work. I also would like to thank R. Jones for his diligent work in preparing the test specimens.

APPENDIX A

Resin Sample Preparation

The resin 3501-6 (obtained from Hercules Inc. Bacchus Utah) was stored in a freezer. Before casting the resin in a sheet mold (made from polished aluminum plates coated with Frekote releasing agent) it was degassed in vacuum by heating the resins till it became sufficiently fluid. Then it was poured into the heated mold. It should be noted that the following curing cycle be carried out without interruption through post cure otherwise the plate cracks easily.

The resin was heat soaked over night (16 hours). The temperature was raised in steps and kept for one hour at each of the following temperatures 100°, 115°, 124°, 130°, 150°C and then the temperature was raised to 177°C and kept at this temperature for 10 hours. From the cured sheets, tensile specimens were machined according to ASTM 638 Type I standard (gage length = 57 mm, width = 13 mm, thickness = 1.8 mm). The sharp edges of the test specimens were smoothed with a fine emery paper to reduce stress concentrations. Small biaxial strain gages were bonded to the tensile specimens which were then dried in vacuum at 105°C for 100 hours and then batches of specimens were exposed for 30 days to the following relative humidities 33, 55, 80 and 100% at 71°C. The samples were then stored at ambient temperature (22°C) in containers with the same corresponding relative humidities.

Mechanical Properties

The initial Young's modulus and Poisson's Ratio were determined at low strains with biaxial strain gages from which the initial shear modulus was calculated. The samples were equilibrated for 4 minutes in the environmental test chamber at the respective temperature by forced air circulation before the load was applied. This time was determined to be sufficient for a temperature equilibration through the sample thickness. (The strain gages on the samples that were exposed to 100% RH debonded easily, and therefore, were useless for the measurement). The total stress strain curves were obtained with an Instron strain gage extensometer (which is less sensitive at low strains). Some additional observations concerning the stress strain measurements are described in the Discussion section.

The total stress-strain curves were measured at a cross head speed of 0.127 cm (.05 inch) per second, while the measurements with the bonded strain gages were done with incremental load increases. The strains were obtained from a digital microstrain gage indicator.

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